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# (2*E*)-1-(4-Methylphenyl)-3-(4-nitrophenyl)prop-2-en-1-one

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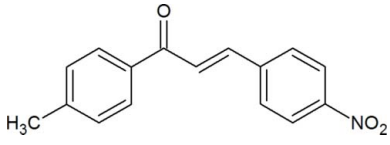
Key indicators: single-crystal X-ray study; $T$ = 203 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.055; $wR$ factor = 0.162; data-to-parameter ratio = 12.4.

The title compound, $C_{16}H_{13}NO_3$, crystallizes with two nearly planar independent molecules in the asymmetric unit. The molecules exist as pseudo-inversion-related pairs and each of the independent molecules forms sheets approximately parallel to the *ab* plane which are alternately stacked along the *c* axis. The crystal structure is stabilized by C—H···O intermolecular hydrogen-bonding interactions.

## Related literature

For related structures, see: Yathirajan *et al.* (2007); Harrison *et al.* (2006); Patil *et al.* (2006). For related literature, see: Dhar *et al.* (1981); Opletalova & Sedivy *et al.* (1999); Sarojini *et al.* (2006).




## Experimental

### Crystal data

$C_{16}H_{13}NO_3$
$M_r$ = 267.27
Monoclinic, *Pc*
*a* = 5.97300 (1) Å
*b* = 15.0731 (5) Å
*c* = 14.6768 (4) Å
$\beta$ = 95.785 (2)°
$V$ = 1314.65 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 203 K
0.47 × 0.41 × 0.29 mm

### Data collection

Oxford Diffraction Gemini R diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
$T_{min}$ = 0.955, $T_{max}$ = 1.000 (expected range = 0.929–0.973)
37695 measured reflections
4496 independent reflections
2661 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.051

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.162
$S$ = 1.25
4496 reflections
363 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C6*A*—H6*A*···O2*B*$^{i}$ | 0.94 | 2.56 | 3.144 (4) | 120 |
| C6*B*—H6*B*···O2*A*$^{ii}$ | 0.94 | 2.57 | 3.409 (4) | 148 |
| C7*B*—H7*D*···O3*B*$^{iii}$ | 0.97 | 2.48 | 3.361 (5) | 151 |
| C16*A*—H16*A*···O1*B*$^{iv}$ | 0.94 | 2.57 | 3.401 (4) | 148 |

Symmetry codes: (i) $x-1, -y, z-\frac{1}{2}$; (ii) $x+1, -y+1, z+\frac{1}{2}$; (iii) $x, y+1, z$; (iv) $x+1, y, z$.

Data collection: *CrysAlisPro* (Oxford Diffraction, 2007); cell refinement: *CrysAlisPro*; data reduction: *CrysAlisPro*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2427).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o3680 [ **doi:10.1107/S1600536807037063** ]

# (2*E*)-1-(4-Methylphenyl)-3-(4-nitrophenyl)prop-2-en-1-one

**R. J. Butcher, J. P. Jasinski, H. S. Yathirajan, K. Veena and B. Narayana**

## Comment

Chalcone is an aromatic ketone that forms a central core for a variety of important biological compounds, which are known collectively as chalcones. Chalcones and the corresponding heterocyclic analogs are valuable intermediates in organic synthesis and show numerous biological effects. The non-linear optical (NLO) effects in organic molecules originates from a strong donor–acceptor intermolecular interaction, a delocalized π electron system and also the ability to crystallize in non-centro symmetric space groups. Chalcones are finding applications as organic non-linear optical materials due to their good SHG conversion efficiencies. Herein we report the synthesis and crystal structure of a new chalcone, the title compound.

The asymmetric unit of the title compound contains two independent molecules A and B both of which are shown in Fig. 1. The C9—C8—C1—C6 and C9—C10—C11—C12 torsion angles [(A) −0.8 (5), 6.4 (5)°; (B) 5.2 (4), −8.2 (5)°] indicate that the 4-methylphenyl and 4-nitrophenyl groups are slightly twisted with respect to the central O1/C8—C11 plane.

In the crystal structure, the independent molecues exist as a pseudo inversion-related pair with the centroids of the 4-methylphenyl and 4-nitrophenyl rings separated by a distance of 3.7550 (17) Å, indicating π-π stacking interaction. Crystal packing shows each of these independent molecules form sheets approximately parallel to the *ab* plane (Fig. 2). The sheets formed by these molecules are alternatively stacked along the *c* axis and are cross-linked by C—H···O intermolecular hydrogen-bonding interactions (Table 1).

## Experimental

4-Nitrobenzaldehyde (1.81 g, 0.01 mol) in ethanol (50 ml) was mixed with 1-(4-methyl phenyl) ethanone (1.34 ml, 0.01 mol) and the mixture was treated with 10 ml of 10% KOH. The reaction mixture was then kept for constant stirring. The solid precipitate obtained was filtered, washed with ethanol and dried. The crystal growth was carried out in acetone solvent by the slow evaporation technique (m.p. 435 K). Analysis found: C 71.78, H 4.83, N 5.18%; $C_{16}H_{13}NO_3$ requires: C 71.90, H 4.90, N 5.24%.

## Refinement

All H atoms were refined using a riding model with C—H = 0.94–0.97 Å, and $U_{iso}$(H) = 1.18–1.50$U_{eq}$(C). In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

## Figures

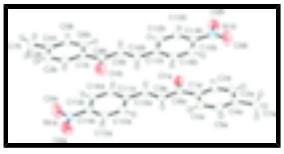

Fig. 1. A view of the two independent molecules (A and B) forming the asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

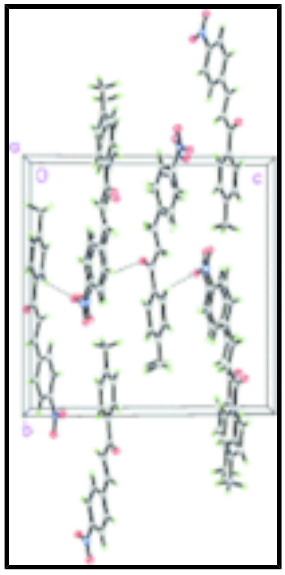

Fig. 2. The crystal packing of the title compound, viewed down the *a* axis. Dashed lines indicate C—H···O hydrogen bonds.

**(2E)-1-(4-Methylphenyl)-3-(4-nitrophenyl)prop-2-en-1-one**

*Crystal data*

| | |
|---|---|
| $C_{16}H_{13}NO_3$ | $F_{000}$ = 560 |
| $M_r$ = 267.27 | $D_x$ = 1.350 Mg m$^{-3}$ |
| Monoclinic, *Pc* | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: P -2yc | Cell parameters from 10389 reflections |
| *a* = 5.97300 (1) Å | θ = 4.6–32.4º |
| *b* = 15.0731 (5) Å | μ = 0.09 mm$^{-1}$ |
| *c* = 14.6768 (4) Å | *T* = 203 K |
| β = 95.785 (2)º | Block, colourless |
| *V* = 1314.65 (6) Å$^3$ | 0.47 × 0.41 × 0.29 mm |
| *Z* = 4 | |

*Data collection*

| | |
|---|---|
| Oxford Diffraction Gemini R diffractometer | 4496 independent reflections |
| Radiation source: fine-focus sealed tube | 2661 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.051 |
| *T* = 203 K | $\theta_{max}$ = 32.5º |
| φ and ω scans | $\theta_{min}$ = 4.6º |
| Absorption correction: multi-scan (CrysAlisRED; Oxford Diffraction, 2007) | *h* = −8→9 |
| $T_{min}$ = 0.955, $T_{max}$ = 1.000 | *k* = −22→21 |
| 37695 measured reflections | *l* = −21→22 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.055$ | $w = 1/[\sigma^2(F_o^2) + (0.0728P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.162$ | $(\Delta/\sigma)_{max} = 0.001$ |
| $S = 1.25$ | $\Delta\rho_{max} = 0.23$ e Å$^{-3}$ |
| 4496 reflections | $\Delta\rho_{min} = -0.23$ e Å$^{-3}$ |
| 363 parameters | Extinction correction: none |
| 2 restraints | |
| Primary atom site location: structure-invariant direct methods | |
| Secondary atom site location: difference Fourier map | |

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on $F^2$, conventional *R*-factors *R* are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2$sigma($F^2$) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1A | 0.5810 (4) | 0.15276 (19) | 0.3609 (2) | 0.0598 (7) |
| O2A | −0.5435 (4) | 0.5503 (2) | 0.1891 (2) | 0.0638 (7) |
| O3A | −0.2876 (5) | 0.6415 (2) | 0.2422 (2) | 0.0717 (9) |
| N1A | −0.3572 (5) | 0.5665 (2) | 0.22697 (19) | 0.0434 (7) |
| C1A | 0.3401 (6) | 0.0319 (3) | 0.3272 (2) | 0.0377 (7) |
| C2A | 0.5097 (5) | −0.0293 (3) | 0.3529 (2) | 0.0434 (8) |
| H2A | 0.6549 | −0.0089 | 0.3732 | 0.052* |
| C3A | 0.4681 (6) | −0.1196 (3) | 0.3492 (3) | 0.0501 (9) |
| H3A | 0.5850 | −0.1594 | 0.3676 | 0.060* |
| C4A | 0.2560 (5) | −0.1524 (2) | 0.3185 (2) | 0.0406 (8) |
| C5A | 0.0874 (6) | −0.0926 (3) | 0.2929 (2) | 0.0452 (9) |
| H5A | −0.0568 | −0.1136 | 0.2718 | 0.054* |
| C6A | 0.1261 (5) | −0.0018 (2) | 0.2977 (2) | 0.0431 (8) |
| H6A | 0.0072 | 0.0376 | 0.2809 | 0.052* |
| C7A | 0.2110 (7) | −0.2512 (3) | 0.3134 (3) | 0.0546 (9) |
| H7A | 0.0911 | −0.2631 | 0.2654 | 0.082* |

| | | | | |
|---|---|---|---|---|
| H7B | 0.1665 | −0.2717 | 0.3716 | 0.082* |
| H7C | 0.3464 | −0.2820 | 0.3001 | 0.082* |
| C8A | 0.3919 (5) | 0.1278 (2) | 0.3334 (2) | 0.0394 (7) |
| C9A | 0.2096 (5) | 0.1932 (3) | 0.3060 (2) | 0.0403 (8) |
| H9A | 0.0735 | 0.1738 | 0.2746 | 0.048* |
| C10A | 0.2392 (5) | 0.2787 (2) | 0.3258 (2) | 0.0382 (7) |
| H10A | 0.3773 | 0.2948 | 0.3579 | 0.046* |
| C11A | 0.0756 (5) | 0.3511 (2) | 0.30221 (19) | 0.0349 (7) |
| C12A | −0.1442 (5) | 0.3358 (2) | 0.2625 (2) | 0.0400 (7) |
| H12A | −0.1949 | 0.2772 | 0.2523 | 0.048* |
| C13A | −0.2877 (5) | 0.4058 (3) | 0.2381 (2) | 0.0408 (8) |
| H13A | −0.4348 | 0.3957 | 0.2110 | 0.049* |
| C14A | −0.2081 (5) | 0.4915 (2) | 0.2549 (2) | 0.0371 (7) |
| C15A | 0.0033 (5) | 0.5092 (2) | 0.2955 (2) | 0.0407 (7) |
| H15A | 0.0516 | 0.5679 | 0.3069 | 0.049* |
| C16A | 0.1458 (5) | 0.4373 (2) | 0.3197 (2) | 0.0404 (7) |
| H16A | 0.2914 | 0.4479 | 0.3482 | 0.048* |
| O1B | −0.3940 (4) | 0.40110 (19) | 0.4712 (2) | 0.0581 (7) |
| O2B | 0.7162 (4) | −0.0017 (2) | 0.6455 (2) | 0.0614 (8) |
| O3B | 0.4447 (5) | −0.0907 (2) | 0.6053 (2) | 0.0647 (8) |
| N1B | 0.5230 (5) | −0.0162 (2) | 0.61432 (18) | 0.0430 (7) |
| C1B | −0.1587 (5) | 0.5225 (2) | 0.51618 (18) | 0.0326 (6) |
| C2B | −0.3283 (6) | 0.5826 (3) | 0.4924 (2) | 0.0404 (8) |
| H2B | −0.4714 | 0.5618 | 0.4695 | 0.048* |
| C3B | −0.2924 (5) | 0.6731 (2) | 0.5014 (2) | 0.0444 (8) |
| H3B | −0.4124 | 0.7125 | 0.4862 | 0.053* |
| C4B | −0.0826 (5) | 0.7064 (3) | 0.5323 (2) | 0.0399 (8) |
| C5B | 0.0894 (5) | 0.6454 (2) | 0.5563 (2) | 0.0416 (7) |
| H5B | 0.2329 | 0.6662 | 0.5786 | 0.050* |
| C6B | 0.0532 (5) | 0.5552 (3) | 0.5479 (2) | 0.0391 (8) |
| H6B | 0.1723 | 0.5155 | 0.5638 | 0.047* |
| C7B | −0.0428 (6) | 0.8042 (3) | 0.5426 (3) | 0.0533 (9) |
| H7D | 0.1068 | 0.8146 | 0.5730 | 0.080* |
| H7E | −0.1538 | 0.8296 | 0.5789 | 0.080* |
| H7F | −0.0557 | 0.8319 | 0.4826 | 0.080* |
| C8B | −0.2056 (5) | 0.4258 (3) | 0.5051 (2) | 0.0378 (8) |
| C9B | −0.0307 (5) | 0.3598 (3) | 0.5351 (2) | 0.0411 (8) |
| H9B | 0.1033 | 0.3785 | 0.5688 | 0.049* |
| C10B | −0.0609 (5) | 0.2744 (2) | 0.5152 (2) | 0.0375 (7) |
| H10B | −0.1970 | 0.2588 | 0.4812 | 0.045* |
| C11B | 0.0964 (5) | 0.2023 (2) | 0.54079 (19) | 0.0353 (7) |
| C12B | 0.3171 (5) | 0.2161 (2) | 0.5814 (2) | 0.0416 (8) |
| H12B | 0.3699 | 0.2743 | 0.5926 | 0.050* |
| C13B | 0.4571 (5) | 0.1451 (2) | 0.6050 (2) | 0.0407 (7) |
| H13B | 0.6043 | 0.1546 | 0.6324 | 0.049* |
| C14B | 0.3778 (5) | 0.0600 (2) | 0.5879 (2) | 0.0358 (7) |
| C15B | 0.1615 (5) | 0.0439 (3) | 0.5465 (2) | 0.0422 (8) |
| H15B | 0.1097 | −0.0143 | 0.5351 | 0.051* |
| C16B | 0.0267 (5) | 0.1153 (2) | 0.5227 (2) | 0.0399 (7) |

| | | | | |
|---|---|---|---|---|
| H16B | −0.1182 | 0.1053 | 0.4932 | 0.048* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1A | 0.0407 (13) | 0.0393 (16) | 0.0947 (19) | −0.0026 (11) | −0.0155 (12) | −0.0039 (13) |
| O2A | 0.0416 (15) | 0.062 (2) | 0.0841 (18) | 0.0046 (12) | −0.0106 (12) | 0.0084 (15) |
| O3A | 0.0562 (16) | 0.0420 (18) | 0.112 (2) | 0.0054 (14) | −0.0137 (15) | 0.0037 (17) |
| N1A | 0.0383 (15) | 0.0412 (19) | 0.0508 (15) | 0.0056 (13) | 0.0050 (12) | 0.0063 (13) |
| C1A | 0.0336 (16) | 0.039 (2) | 0.0403 (15) | −0.0015 (14) | 0.0033 (12) | −0.0021 (14) |
| C2A | 0.0300 (16) | 0.039 (2) | 0.0592 (19) | 0.0030 (14) | −0.0058 (13) | −0.0014 (16) |
| C3A | 0.0412 (19) | 0.043 (2) | 0.064 (2) | 0.0060 (17) | −0.0044 (15) | 0.0048 (18) |
| C4A | 0.0415 (18) | 0.035 (2) | 0.0452 (16) | 0.0005 (14) | 0.0045 (13) | 0.0006 (13) |
| C5A | 0.0380 (18) | 0.041 (2) | 0.0547 (18) | −0.0036 (16) | −0.0031 (14) | −0.0006 (17) |
| C6A | 0.0342 (17) | 0.035 (2) | 0.0581 (19) | 0.0046 (14) | −0.0037 (13) | −0.0031 (15) |
| C7A | 0.063 (2) | 0.037 (2) | 0.064 (2) | −0.0034 (19) | 0.0048 (17) | 0.0001 (17) |
| C8A | 0.0384 (16) | 0.0322 (19) | 0.0460 (16) | 0.0023 (14) | −0.0042 (12) | 0.0004 (13) |
| C9A | 0.0396 (18) | 0.033 (2) | 0.0454 (17) | −0.0021 (15) | −0.0077 (13) | −0.0009 (14) |
| C10A | 0.0397 (16) | 0.0355 (19) | 0.0385 (14) | 0.0016 (14) | −0.0003 (11) | −0.0018 (13) |
| C11A | 0.0342 (16) | 0.0344 (19) | 0.0361 (14) | 0.0011 (13) | 0.0035 (11) | 0.0015 (13) |
| C12A | 0.0357 (16) | 0.036 (2) | 0.0477 (16) | −0.0062 (14) | 0.0012 (12) | −0.0008 (14) |
| C13A | 0.0304 (16) | 0.043 (2) | 0.0489 (17) | −0.0010 (15) | 0.0036 (13) | −0.0004 (15) |
| C14A | 0.0365 (17) | 0.036 (2) | 0.0387 (15) | 0.0028 (14) | 0.0059 (12) | 0.0025 (12) |
| C15A | 0.0383 (17) | 0.0344 (19) | 0.0497 (17) | −0.0026 (14) | 0.0053 (13) | 0.0016 (14) |
| C16A | 0.0380 (16) | 0.039 (2) | 0.0434 (15) | −0.0029 (14) | −0.0007 (12) | −0.0038 (14) |
| O1B | 0.0446 (14) | 0.0446 (16) | 0.0805 (17) | −0.0035 (11) | −0.0155 (11) | −0.0055 (13) |
| O2B | 0.0337 (14) | 0.059 (2) | 0.0889 (18) | 0.0037 (11) | −0.0074 (12) | 0.0012 (15) |
| O3B | 0.0501 (15) | 0.0375 (17) | 0.103 (2) | 0.0023 (13) | −0.0084 (14) | 0.0014 (15) |
| N1B | 0.0379 (16) | 0.0441 (19) | 0.0466 (14) | 0.0029 (13) | 0.0023 (11) | 0.0020 (13) |
| C1B | 0.0334 (15) | 0.0319 (18) | 0.0324 (13) | 0.0010 (12) | 0.0033 (11) | 0.0017 (11) |
| C2B | 0.0330 (16) | 0.040 (2) | 0.0464 (16) | −0.0027 (14) | −0.0040 (12) | 0.0011 (15) |
| C3B | 0.0374 (17) | 0.039 (2) | 0.0555 (18) | 0.0049 (14) | −0.0016 (13) | 0.0005 (15) |
| C4B | 0.0392 (17) | 0.038 (2) | 0.0431 (16) | −0.0012 (14) | 0.0075 (13) | 0.0024 (14) |
| C5B | 0.0294 (15) | 0.044 (2) | 0.0506 (17) | 0.0007 (14) | −0.0016 (12) | −0.0020 (15) |
| C6B | 0.0310 (16) | 0.039 (2) | 0.0463 (17) | −0.0012 (14) | 0.0000 (12) | −0.0025 (15) |
| C7B | 0.051 (2) | 0.034 (2) | 0.074 (2) | −0.0020 (17) | 0.0044 (16) | −0.0014 (17) |
| C8B | 0.0429 (18) | 0.036 (2) | 0.0335 (14) | −0.0028 (14) | −0.0014 (12) | −0.0014 (13) |
| C9B | 0.0372 (17) | 0.040 (2) | 0.0441 (16) | 0.0010 (14) | −0.0043 (12) | −0.0034 (14) |
| C10B | 0.0359 (16) | 0.036 (2) | 0.0399 (15) | −0.0008 (14) | 0.0022 (12) | 0.0054 (14) |
| C11B | 0.0387 (16) | 0.0319 (18) | 0.0354 (14) | 0.0000 (13) | 0.0040 (11) | 0.0009 (12) |
| C12B | 0.0387 (17) | 0.0340 (18) | 0.0518 (18) | −0.0049 (14) | 0.0035 (13) | −0.0021 (14) |
| C13B | 0.0352 (16) | 0.041 (2) | 0.0452 (16) | −0.0061 (14) | −0.0004 (12) | −0.0043 (14) |
| C14B | 0.0323 (16) | 0.0385 (19) | 0.0364 (14) | 0.0010 (14) | 0.0022 (12) | 0.0000 (13) |
| C15B | 0.0373 (18) | 0.037 (2) | 0.0509 (18) | −0.0033 (14) | −0.0028 (13) | −0.0052 (15) |
| C16B | 0.0334 (16) | 0.037 (2) | 0.0480 (16) | −0.0073 (14) | −0.0030 (12) | −0.0027 (14) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1A—C8A | 1.220 (4) | O1B—C8B | 1.240 (4) |

| | | | |
|---|---|---|---|
| O2A—N1A | 1.218 (4) | O2B—N1B | 1.217 (4) |
| O3A—N1A | 1.217 (4) | O3B—N1B | 1.219 (4) |
| N1A—C14A | 1.472 (4) | N1B—C14B | 1.468 (5) |
| C1A—C2A | 1.394 (5) | C1B—C2B | 1.378 (5) |
| C1A—C6A | 1.403 (5) | C1B—C6B | 1.394 (5) |
| C1A—C8A | 1.479 (5) | C1B—C8B | 1.490 (5) |
| C2A—C3A | 1.383 (5) | C2B—C3B | 1.385 (5) |
| C2A—H2A | 0.94 | C2B—H2B | 0.94 |
| C3A—C4A | 1.392 (5) | C3B—C4B | 1.384 (5) |
| C3A—H3A | 0.94 | C3B—H3B | 0.94 |
| C4A—C5A | 1.376 (5) | C4B—C5B | 1.397 (5) |
| C4A—C7A | 1.513 (5) | C4B—C7B | 1.499 (5) |
| C5A—C6A | 1.388 (5) | C5B—C6B | 1.380 (5) |
| C5A—H5A | 0.94 | C5B—H5B | 0.94 |
| C6A—H6A | 0.94 | C6B—H6B | 0.94 |
| C7A—H7A | 0.97 | C7B—H7D | 0.97 |
| C7A—H7B | 0.97 | C7B—H7E | 0.97 |
| C7A—H7C | 0.97 | C7B—H7F | 0.97 |
| C8A—C9A | 1.494 (5) | C8B—C9B | 1.476 (5) |
| C9A—C10A | 1.328 (5) | C9B—C10B | 1.329 (5) |
| C9A—H9A | 0.94 | C9B—H9B | 0.94 |
| C10A—C11A | 1.482 (5) | C10B—C11B | 1.461 (5) |
| C10A—H10A | 0.94 | C10B—H10B | 0.94 |
| C11A—C16A | 1.382 (5) | C11B—C16B | 1.394 (5) |
| C11A—C12A | 1.401 (4) | C11B—C12B | 1.407 (5) |
| C12A—C13A | 1.384 (5) | C12B—C13B | 1.380 (5) |
| C12A—H12A | 0.94 | C12B—H12B | 0.94 |
| C13A—C14A | 1.390 (5) | C13B—C14B | 1.381 (5) |
| C13A—H13A | 0.94 | C13B—H13B | 0.94 |
| C14A—C15A | 1.367 (5) | C14B—C15B | 1.393 (5) |
| C15A—C16A | 1.401 (5) | C15B—C16B | 1.367 (5) |
| C15A—H15A | 0.94 | C15B—H15B | 0.94 |
| C16A—H16A | 0.94 | C16B—H16B | 0.94 |
| O2A—N1A—O3A | 123.4 (3) | O2B—N1B—O3B | 123.0 (3) |
| O2A—N1A—C14A | 118.2 (3) | O2B—N1B—C14B | 118.1 (3) |
| O3A—N1A—C14A | 118.4 (3) | O3B—N1B—C14B | 118.9 (3) |
| C2A—C1A—C6A | 117.3 (3) | C2B—C1B—C6B | 118.1 (3) |
| C2A—C1A—C8A | 119.2 (3) | C2B—C1B—C8B | 119.4 (3) |
| C6A—C1A—C8A | 123.5 (3) | C6B—C1B—C8B | 122.5 (3) |
| C3A—C2A—C1A | 121.2 (3) | C1B—C2B—C3B | 121.4 (3) |
| C3A—C2A—H2A | 119.4 | C1B—C2B—H2B | 119.3 |
| C1A—C2A—H2A | 119.4 | C3B—C2B—H2B | 119.3 |
| C2A—C3A—C4A | 121.1 (3) | C2B—C3B—C4B | 121.0 (3) |
| C2A—C3A—H3A | 119.5 | C2B—C3B—H3B | 119.5 |
| C4A—C3A—H3A | 119.5 | C4B—C3B—H3B | 119.5 |
| C5A—C4A—C3A | 118.2 (3) | C3B—C4B—C5B | 117.6 (3) |
| C5A—C4A—C7A | 120.6 (3) | C3B—C4B—C7B | 121.3 (3) |
| C3A—C4A—C7A | 121.1 (3) | C5B—C4B—C7B | 121.1 (3) |
| C4A—C5A—C6A | 121.2 (3) | C6B—C5B—C4B | 121.4 (3) |

| | | | |
|---|---|---|---|
| C4A—C5A—H5A | 119.4 | C6B—C5B—H5B | 119.3 |
| C6A—C5A—H5A | 119.4 | C4B—C5B—H5B | 119.3 |
| C5A—C6A—C1A | 121.0 (3) | C5B—C6B—C1B | 120.5 (3) |
| C5A—C6A—H6A | 119.5 | C5B—C6B—H6B | 119.8 |
| C1A—C6A—H6A | 119.5 | C1B—C6B—H6B | 119.8 |
| C4A—C7A—H7A | 109.5 | C4B—C7B—H7D | 109.5 |
| C4A—C7A—H7B | 109.5 | C4B—C7B—H7E | 109.5 |
| H7A—C7A—H7B | 109.5 | H7D—C7B—H7E | 109.5 |
| C4A—C7A—H7C | 109.5 | C4B—C7B—H7F | 109.5 |
| H7A—C7A—H7C | 109.5 | H7D—C7B—H7F | 109.5 |
| H7B—C7A—H7C | 109.5 | H7E—C7B—H7F | 109.5 |
| O1A—C8A—C1A | 120.2 (3) | O1B—C8B—C9B | 120.3 (3) |
| O1A—C8A—C9A | 120.7 (3) | O1B—C8B—C1B | 119.3 (3) |
| C1A—C8A—C9A | 119.1 (3) | C9B—C8B—C1B | 120.4 (3) |
| C10A—C9A—C8A | 120.3 (3) | C10B—C9B—C8B | 120.7 (3) |
| C10A—C9A—H9A | 119.9 | C10B—C9B—H9B | 119.7 |
| C8A—C9A—H9A | 119.9 | C8B—C9B—H9B | 119.7 |
| C9A—C10A—C11A | 126.4 (3) | C9B—C10B—C11B | 126.5 (3) |
| C9A—C10A—H10A | 116.8 | C9B—C10B—H10B | 116.7 |
| C11A—C10A—H10A | 116.8 | C11B—C10B—H10B | 116.7 |
| C16A—C11A—C12A | 119.1 (3) | C16B—C11B—C12B | 118.0 (3) |
| C16A—C11A—C10A | 117.9 (3) | C16B—C11B—C10B | 118.6 (3) |
| C12A—C11A—C10A | 123.0 (3) | C12B—C11B—C10B | 123.4 (3) |
| C13A—C12A—C11A | 120.8 (3) | C13B—C12B—C11B | 120.6 (3) |
| C13A—C12A—H12A | 119.6 | C13B—C12B—H12B | 119.7 |
| C11A—C12A—H12A | 119.6 | C11B—C12B—H12B | 119.7 |
| C12A—C13A—C14A | 118.1 (3) | C14B—C13B—C12B | 119.1 (3) |
| C12A—C13A—H13A | 120.9 | C14B—C13B—H13B | 120.4 |
| C14A—C13A—H13A | 120.9 | C12B—C13B—H13B | 120.4 |
| C15A—C14A—C13A | 122.9 (3) | C13B—C14B—C15B | 121.8 (3) |
| C15A—C14A—N1A | 118.6 (3) | C13B—C14B—N1B | 119.7 (3) |
| C13A—C14A—N1A | 118.5 (3) | C15B—C14B—N1B | 118.4 (3) |
| C14A—C15A—C16A | 118.1 (3) | C16B—C15B—C14B | 118.1 (3) |
| C14A—C15A—H15A | 121.0 | C16B—C15B—H15B | 121.0 |
| C16A—C15A—H15A | 121.0 | C14B—C15B—H15B | 121.0 |
| C11A—C16A—C15A | 121.0 (3) | C15B—C16B—C11B | 122.3 (3) |
| C11A—C16A—H16A | 119.5 | C15B—C16B—H16B | 118.8 |
| C15A—C16A—H16A | 119.5 | C11B—C16B—H16B | 118.8 |
| C6A—C1A—C2A—C3A | 0.2 (5) | C6B—C1B—C2B—C3B | −1.4 (5) |
| C8A—C1A—C2A—C3A | 179.3 (3) | C8B—C1B—C2B—C3B | −179.9 (3) |
| C1A—C2A—C3A—C4A | 0.8 (5) | C1B—C2B—C3B—C4B | 1.8 (5) |
| C2A—C3A—C4A—C5A | −0.7 (5) | C2B—C3B—C4B—C5B | −1.6 (5) |
| C2A—C3A—C4A—C7A | 179.4 (3) | C2B—C3B—C4B—C7B | −179.7 (3) |
| C3A—C4A—C5A—C6A | −0.2 (5) | C3B—C4B—C5B—C6B | 1.2 (5) |
| C7A—C4A—C5A—C6A | 179.7 (3) | C7B—C4B—C5B—C6B | 179.2 (3) |
| C4A—C5A—C6A—C1A | 1.2 (5) | C4B—C5B—C6B—C1B | −0.9 (5) |
| C2A—C1A—C6A—C5A | −1.2 (5) | C2B—C1B—C6B—C5B | 0.9 (5) |
| C8A—C1A—C6A—C5A | 179.8 (3) | C8B—C1B—C6B—C5B | 179.4 (3) |
| C2A—C1A—C8A—O1A | −0.1 (5) | C2B—C1B—C8B—O1B | 2.9 (4) |

| | | | |
|---|---|---|---|
| C6A—C1A—C8A—O1A | 179.0 (3) | C6B—C1B—C8B—O1B | −175.6 (3) |
| C2A—C1A—C8A—C9A | −179.8 (3) | C2B—C1B—C8B—C9B | −176.4 (3) |
| C6A—C1A—C8A—C9A | −0.8 (5) | C6B—C1B—C8B—C9B | 5.2 (4) |
| O1A—C8A—C9A—C10A | −11.7 (5) | O1B—C8B—C9B—C10B | 9.2 (5) |
| C1A—C8A—C9A—C10A | 168.1 (3) | C1B—C8B—C9B—C10B | −171.5 (3) |
| C8A—C9A—C10A—C11A | 179.2 (3) | C8B—C9B—C10B—C11B | −179.7 (3) |
| C9A—C10A—C11A—C16A | −173.0 (3) | C9B—C10B—C11B—C16B | 173.0 (3) |
| C9A—C10A—C11A—C12A | 6.4 (5) | C9B—C10B—C11B—C12B | −8.2 (5) |
| C16A—C11A—C12A—C13A | 2.1 (4) | C16B—C11B—C12B—C13B | −1.9 (4) |
| C10A—C11A—C12A—C13A | −177.2 (3) | C10B—C11B—C12B—C13B | 179.3 (3) |
| C11A—C12A—C13A—C14A | −0.5 (4) | C11B—C12B—C13B—C14B | 0.2 (4) |
| C12A—C13A—C14A—C15A | −1.1 (5) | C12B—C13B—C14B—C15B | 0.8 (5) |
| C12A—C13A—C14A—N1A | 178.3 (3) | C12B—C13B—C14B—N1B | −178.7 (3) |
| O2A—N1A—C14A—C15A | 178.5 (3) | O2B—N1B—C14B—C13B | −4.7 (4) |
| O3A—N1A—C14A—C15A | −1.0 (4) | O3B—N1B—C14B—C13B | 174.3 (3) |
| O2A—N1A—C14A—C13A | −1.0 (4) | O2B—N1B—C14B—C15B | 175.7 (3) |
| O3A—N1A—C14A—C13A | 179.6 (3) | O3B—N1B—C14B—C15B | −5.3 (4) |
| C13A—C14A—C15A—C16A | 1.1 (5) | C13B—C14B—C15B—C16B | −0.1 (5) |
| N1A—C14A—C15A—C16A | −178.3 (3) | N1B—C14B—C15B—C16B | 179.5 (3) |
| C12A—C11A—C16A—C15A | −2.1 (4) | C14B—C15B—C16B—C11B | −1.7 (5) |
| C10A—C11A—C16A—C15A | 177.3 (3) | C12B—C11B—C16B—C15B | 2.7 (5) |
| C14A—C15A—C16A—C11A | 0.6 (5) | C10B—C11B—C16B—C15B | −178.4 (3) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C6A—H6A···O2B$^{i}$ | 0.94 | 2.56 | 3.144 (4) | 120 |
| C6B—H6B···O2A$^{ii}$ | 0.94 | 2.57 | 3.409 (4) | 148 |
| C7B—H7D···O3B$^{iii}$ | 0.97 | 2.48 | 3.361 (5) | 151 |
| C16A—H16A···O1B$^{iv}$ | 0.94 | 2.57 | 3.401 (4) | 148 |

Symmetry codes: (i) *x*−1, −*y*, *z*−1/2; (ii) *x*+1, −*y*+1, *z*+1/2; (iii) *x*, *y*+1, *z*; (iv) *x*+1, *y*, *z*.

**Fig. 1**

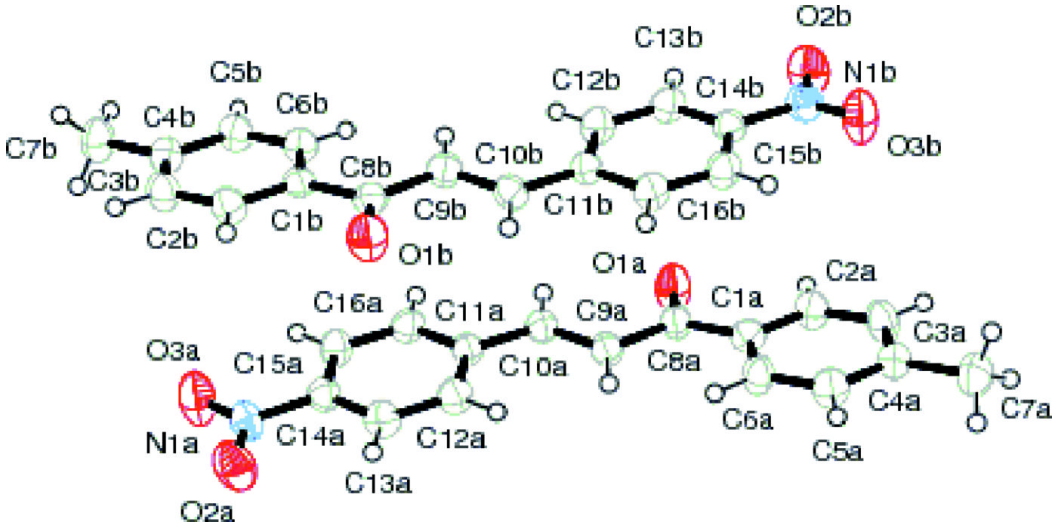
O2b
C13b
N1b
C12b
C14b
C5b
C6b
C4b
C7b
C15b
O3b
C3b
C8b
C10b
C1b
C9b
C11b
C16b
C2b
O1b
O1a
C2a
C16a
C11a
C9a
C1a
C3a
O3a
C15a
C10a
C8a
C4a
C7a
C6a
C14a
C12a
N1a
C5a
C13a
O2a

**Fig. 2**